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PERFORMANCE OF CERAMIC INSULATOR SEALS AT HIGH NEUTRON AND GAMMA FLUENCE LEVELS

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16. Abstract

This report describes an experimental program which was undertaken to evaluate the in-pile performance of a group of commercially available ceramic insulator seals. The ceramic insulator seals were irradiated to thermal neutron fluence levels greater than 10^{20} neutrons/cm^2 and gamma dose rates greater than 10^{14} ergs/g of H_2O. During the irradiation the seals were thermally cycled several times. Neutron radiographic investigation showed that all 24 seals investigated were leaktight to reactor cooling water at 150° F (339 K) and 150 psia (103.4 N/cm^2) throughout the irradiation.

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SUMMARY

An experimental program was undertaken to evaluate the in-pile performance of a group of commercially available ceramic insulator seals. There is an obvious lack of in-pile irradiation information on ceramic insulator seals subjected to thermal nvt levels exceeding 10^{20} neutrons per square centimeter. Also the little information available is for seals exposed to gases. For the application intended, information was needed for ceramic insulators having one side of the seal exposed to hot (150° F, 339 K), deionized water.

In this program, four different types of ceramic insulator seals were investigated. They were made by two different manufacturers, employed different insulator-metal brazes or bonds, and used different materials for their metal parts. All used alumina for the insulator material.

Six ceramic insulators of each of the four types were tested. They were irradiated to thermal nvt levels greater than 10^{20} neutrons per square centimeter and gamma dose levels greater than 10^{14} ergs per gram of water. Before and during irradiation, they were thermally cycled in order to evaluate whether this would cause the ceramic-metal braze joint to fail and thus cause a leak.

The irradiation program showed that the ceramic insulator seals tested could withstand the reported neutron fluence and gamma dose levels, as well as several thermal cycles. Neutron radiography showed no leakage of the 150° F (339 K), 150-psia (103.4-N/cm^2) deionized water through any of the seals.

INTRODUCTION

An instrumented hydraulic multiple-capsule facility (ref. 1) for the Plum Brook Reactor is used to test fuel pins to high burnups. Each capsule depends on a double insulator-seal assembly to bring out a thermocouple lead to a recorder monitoring fuelpin temperature. Before such tests could be undertaken, it was necessary to evaluate the in-pile operation of commercially available insulator seals.

The nature of the facility and tests required that the seals operate successfully to high fluence levels when subjected to at least 6 thermal cycles from 68° to 150° F (293 to 339 K) and with one side of the seal exposed to deionized water. Successful operation of seals was defined as operating without leaking water at 150 psia (103.4 N/cm^2).

Four slightly different types of ceramic insulator seals were selected for test, and six of each type were used to fabricate three double insulator-seal assemblies of each type. The 12 double insulator-seal assemblies were incorporated into six irradiation capsules, which were irradiated simultaneously in the aforementioned facility.

Before irradiation, the 24 ceramic insulator seals were thermally cycled and helium leak-checked. After assembly into 12 double insulator-seal assemblies, they were irradiated to thermal nvt levels greater than 10^{20} neutrons per square centimeter and gamma dose levels greater than 10^{14} ergs per gram of H_2O. They were neutron radiographed periodically during irradiation, and also afterwards, for evidence of water leakage past the insulator seals. Post-irradiation examination showed that all ceramic seals successfully completed the test without trace of water leakage.

The following sections describe the ceramic seals, the seal assemblies, and the test capsule hardware. The methods and results of pre-irradiation testing and post-irradiation examination are discussed.

DESCRIPTION

Ceramic Insulator Seal and Holder

All the ceramic insulator seals tested had the same general appearance (fig. 1(a)). All were approximately 1/4 inch (0.635 cm) in diameter and about $1\frac{1}{2}$ inches (3.80 cm) long. All employed a high-purity alumina cylinder as the insulator, to which is brazed a metal skirt at one end and a metal cap at the other end. The different types were characterized by different brazes and different skirt and cap materials as given in table I.

A satisfactory design for the fixture to which the ceramic insulator seal is welded is shown in figure 1(b). For welding, it was advantageous to machine a thin tube appendage on the fixture to accept the ceramic insulator skirt. All fixtures were made from stainless steel.

It was found that preparation of the seals and the welding techniques used greatly affected the success with which we could obtain leaktight welds. To prepare for the welding, the ceramic insulator and stainless-steel fixture were fitted at the weld point.

The skirt of the ceramic insulator was cleaned with abrasive cloth containing alumina grit to remove any braze material. Both the fixture and the ceramic insulator were cleaned in acetone and dried in air. Then the ceramic insulator was gas tungsten-arc welded (GTAW) to the fixture, as shown in figure 1(c). Also the opposite end of the ceramic insulator was closed off with a looped wire (fig. 1(c)).

Double Ceramic Insulator Seals

Each of the 24 ceramic insulator seals with fixture (fig. 1(c)) were helium leak-checked, thermally cycled, and leak-checked again. Then each two identical-type seals were electron-beam-welded together, as shown in figure 2, to form a double ceramic insulator seal with an evacuated chamber between the two seals. Twelve of these double insulator-seal assemblies were fabricated from the 24 ceramic insulator seals.

Test Capsules and Assembly

Two of these double insulator-seal assemblies were then joined and protected by stainless-steel perforated tubes to form a capsule which could conveniently be irradiated in the multiple-capsule facility. The two double insulator-seal assemblies and perforated tubes are shown in figure 3. These parts are welded together to produce the test capsule shown in figure 4. It is 0.690 inch (1.75 cm) in diameter and 7.57 inches (19.23 cm) long.

Before the tubes and chambers were welded, three small wires were attached to the loop on the ends of several of the ceramic insulators. These wires were made of nickel, stainless steel, titanium, and a cobalt-aluminum alloy. These wires would be activated during irradiation to permit evaluation of thermal flux as well as of fast and intermediate fluxes.

Six of these test capsules were then assembled to form a multiple-capsule test assembly for irradiation in the multiple-capsule facility (ref. 1) in the Plum Brook Reactor.

METHODS AND RESULTS

Pre-Irradiation Tests

Thermal cycling of single insulator seals. - Before irradiation, the 24 single ceramic seal-and-holder units (fig. 1(c)) were thermally cycled. The purpose was to determine if the seals might have a tendency to fail from thermal cycling rather than from irradiation. Each ceramic seal-holder unit was connected to a helium supply and a helium leak detector, as shown in figure 5. The helium pressure on one side of the ceramic insulator was maintained at a pressure slightly above atmospheric. The leak detector measured any helium that might leak through the ceramic-to-metal braze at either end of the seal unit. Each ceramic insulator seal was first leak-checked while immersed in water at approximately 68° F (293 K). The leak rate was recorded. Then the ceramic insulator was immersed in water at approximately 200° F (367 K) and its leak rate recorded again. The ceramic insulator was then thermally cycled 15 times by switching from the 68° F (293 K) water to the 200° F (367 K) water. Then the ceramic insulator was leak-checked in water at 68° F (293 K) and at 200° F (367 K) and both leak rates were recorded. Each time the ceramic insulator was placed in water at either temperature it was left in for at least 2 minutes before leak detector readings were taken. The results of the thermal cycling are given in table II. The leak rates of all the ceramic insulators was small both before and after thermal cycling. The immersion of the seals in hot water had little effect on the leak rate. The leak rate of the seals changed very little after 15 thermal cycles and again no significant change occurred when the insulator was immersed in the hot water.

The magnitude of the leak rates in table II (approx. 10^{-8} ft^3/hr of He at STP) was well below that necessary to leak water. Leak rates of approximately 10^{-5} cubic feet per hour of helium at standard temperature and pressure, or less, should show no noticeable water leaks (ref. 2).

Leak testing of double insulator seals. - Once a pair of ceramic insulator seals were welded together to form a double insulator-seal unit (fig. 2), the evacuated chamber between the seals was leak-checked. The sample was first placed in a container and pressurized with helium to 50 psi (34.4 N/cm^2) and left for 1/2 hour. Then the sample was removed from that container and placed in one connected to the leak detector, where it was checked for helium leaks from the evacuated chamber. This was done at ambient conditions.

A large leak might release all helium before it could be detected on the leak detector. Therefore a hydrostatic test was conducted to check the chamber for large leaks. The chambers were placed in a tank of water, pressurized to 150 psi (103.4 N/cm^2) and left overnight. The chambers were neutron radiographed before and after the immersion. Any water in the chamber would clearly show on the radiographs. Figure 6 is a neutron radiograph of the capsule assembly before it underwent the hydrostatic test. Figure 7 is a neutron radiograph of the capsule assembly after the hydrostatic test. Both neutron radiographs show no trace of water in the evacuated chambers. If it were present, water would show as a white image on the neutron radiographs.

Irradiation

The multiple-capsule test assembly was then irradiated for about 960 hours, which exposed the ceramic insulator seals to a thermal neutron fluence greater than 10^{20} neutrons per square centimeter and a gamma dose greater than 10^{14} ergs per gram of H_2O. While the capsule assembly was undergoing irradiation, it was thermally cycled six times between 70° and 150° F (294 and 339 K).

The test assembly was removed from the reactor and neutron radiographed after each 200 to 300 hours of irradiation until the test was completed. The radiographs were taken to show any evidence of water leaks past the seals into the evacuated chambers. The results are discussed in the next section. The test assembly was then removed to the hot lab for post-irradiation examination.

Post-Irradiation Examination

Neutron radiographs. - As previously mentioned, neutron radiographic surveillance was used mainly because it is an acceptable and relatively easy method to detect the presence of water in the evacuated chambers (ref. 3). The radiographs taken are shown in figures 6 to 10. If the chamber contains no water, the neutrons passing through it will not be affected and as a result the photograph will be exposed in that area (appears dark in figs. 6 to 10). If the chamber has water in it, the neutrons passing through the chamber will be affected (either absorbed or scattered) and as a result the photograph will not be exposed in that area (would appear as white in figs. 6 to 10). The neutron radiographs taken after 250, 550, and 960 hours of irradiation (figs. 8 to 10) showed no evidence of water leakage through the seals and into the chambers.

Visual inspection. - After the ceramic insulators had been irradiated for 960 hours, the capsule assembly was transferred to the hot lab for visual inspection. Three of the individual capsules were removed from the holder assembly. The ceramic insulators in these three capsules included some of each of the four types of seals irradiated. These three capsules also covered the maximum, average, and minimum irradiation doses in the holder assembly. The other three capsules were left in the holder assembly for possible additional irradiation. The individual ceramic insulators were exposed for visual inspection by cutting the perforated connecting tubes (fig. 3) with a tube cutter. Most of these cuts were made at the weld joint between the perforated tube and the double ceramic insulator chamber to provide the greatest exposure of the insulators.

During the initial disassembly from the holder, the ceramic on seal 21 was found to be broken. After reviewing the final neutron radiograph and later noting that there was water in the double ceramic insulator chamber, it was concluded that the ceramic must have been broken during transfer to the hot lab.

Each exposed ceramic insulator was visually inspected under a stereo microscope at 0.66×5, 1×5, and 2×5 magnification. Photographs were taken as a record. Typical photographs are shown in figures 11 to 14. The results of the visual inspection are summarized in table III.

Surface cracking or checking was noted on all ceramics with surface glazing. Several ceramic insulators were coated with dye penetrant to determine the depth of the cracking. The insulators were then broken by bending and color photographs were taken of the broken cross sections to measure the penetration of the dye. This penetration ranges between 12 and 14 percent of the thickness of the ceramic cylinder. It was noted that all ceramic insulators broke just inside the metal skirt (point of maximum stress) when the breaking force was applied at the metal cap end. These breaks showed the normal tension-compression-type break expected.

Some of the ceramic insulators also showed signs of corrosion in the area where the flux wires were attached (figs. 12 and 14). Corrosion appeared on those ceramic insulators which had some of their parts made from an iron-based alloy, even though all ceramic insulators had a 0.0008-inch (0.002-cm) nickel coating electroplated on their metal parts. It is believed that the welding of the eye for the flux measuring wires caused vaporization of the nickel plating, thus exposing the iron-based alloy material. The exposed material was corroded by its exposure to water.

Neutron fluence and gamma dose evaluation. - The 0.5 percent Co-Al, Ni, Ti, and stainless-steel flux wires were removed from the ends of the ceramic insulators and counted. The neutron fluence was determined and is summarized in table III for each insulator position. The thermal neutron fluences for seals 8, 16, and 24 were estimated from previous flux curves for the test facility normalized to the measurements on the other insulators. The gamma dose for each seal was based upon Plum Brook Mockup Reactor measurements for the test facility (ref. 4).

CONCLUSIONS

As a result of the tests undertaken to evaluate the in-pile performance of a group of commercially available ceramic insulator seals, it was found that

1. All commercially available ceramic insulator seals tested can withstand thermal neutron fluence levels greater than 10^{20} neutrons per square centimeter and gamma doses of 10^{14} ergs per gram of H_2O.

2. None of the ceramic seals tested showed any evidence of water leakage (150° F (339 K) and 150-psia (103.4-N/cm^2) deionized water) past the seals after 960 hours of irradiation.

3. The seals did show some visible damage after the irradiation test. This damage appeared as surface checking of the glazing on the alumina insulators. The checking penetration was only about 12 to 14 percent of the alumina wall thickness.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 22, 1970,
120-27.

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TABLE I. - CHARACTERIZATION OF CERAMIC INSULATORS[a]

Type	Braze	Skirt material	Cap material	Insulator
A	Silver[b]	SS-304	SS-304	96 Percent Al_2O_3
B	Silver[b]	Kovar[c] (nickel plated)	Kovar[c] (nickel plated)	96 Percent Al_2O_3
C	Silver[b]	Nickel	Nickel	86 Percent Al_2O_3[d]
D	Copper[b]	Nickel	Nickel	86 Percent Al_2O_3[d]

[a]All of the ceramic insulator seals were glazed except for one pair of type D insulators.

[b]Braze was nickel plated.

[c]Composition of Kovar in percent: 53.7 Fe, 29.0 Ni, 17.0 Co, and 0.3 Mn.

[d]Insulator also contained 10 percent SiO_2, 2 percent MgO, and 2 percent CaO and BaO.

TABLE II. - THERMAL CYCLING RESULTS

Seal	Braze material	Skirt material	Leak rate before cycling, ft^3/hr of He at STP		Leak rate after 15 cycles, ft^3/hr of He at STP	
			200° F (367 K)	68° F (293 K)	200° F (367 K)	68° F (293 K)
1	Silver	SS-304	2.0×10^{-9}	1.2×10^{-9}	9.0×10^{-9}	9.2×10^{-9}
2			1.7	2.0	4.0	3.0
3			1.6	1.1	4.0	2.6
4			.7	.6	6.0	5.8
5		Kovar[a]	8.0	8.0	10.0	6.0
6		Kovar[a]	.9	1.0	1.0	1.0
7		SS-304	3.1	1.4	2.5	1.2
8		SS-304	2.6	1.4	2.2	1.4
9		Kovar[a]	2.1	1.2	1.9	1.5
10			1.6	1.5	2.2	3.8
11			10.0	9.0	4.5	10.0
12			10.0	14.0	3.5	6.0
13		Nickel	1.2	.6	1.4	1.8
14			2.6	6.0	1.3	6.0
15			10.0	10.0	10.0	10.0
16			3.0	4.5	3.3	2.3
17	Copper		2.0	1.4	1.5	1.2
18			1.7	1.2	1.4	1.2
19			4.0	2.0	2.5	4.1
20			1.6	1.4	1.6	2.9
21	Silver		.4	2.0	2.4	2.0
22	Silver		1.6	1.4	1.8	6.7
23	Copper		4.0	1.8	3.4	3.5
24	Copper		1.8	1.4	3.4	6.4

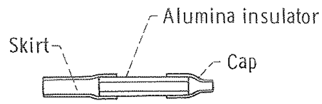
[a]Composition of Kovar in percent: 53.7 Fe, 29.0 Ni, 17.0 Co, and 0.3 Mn.
[b]Alumina insulator of this seal was not glazed.

TABLE III. - SUMMARY OF POST-IRRADIATION EXAMINATION

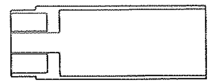
Ceramic insulator seals 1 to 4, 9 to 12, and 17 to 20 were not examined visually. It is intended to run these seals to higher nvt's in the future.

Seal number	Thermal neutron fluence, neutrons per cm^2	Fast neutron fluence, neutrons per cm^2		Gamma dose, ergs per g of H_2O (a)	Ceramic inspection results	Appearance of ceramic-to-metal braze joint
		>0.1 MeV	>1.0 MeV			
[b]5	[c]8.4×10^{20}	[c]9.1×10^{19}	5.5×10^{19}	1.6×10^{14}	Small surface checks, ~0.02 inch by 0.02 inch square shapes; maximum dye penetration, about 12 percent; very difficult to break; see figure 11	Clean and bright, no indication of corrosion (see fig. 11)
6 7	[c]7.5×10^{20} [c]6.8	[c]8.1×10^{19} [c]7.9	4.9×10^{19} 4.8	1.4×10^{14}	Surface checks similar to seal 5; no dye check	
8	[d]5.5×10^{20}	---------	--------	1.1×10^{14}	Surface checks similar to seal 5; maximum dye penetration about 14 percent; very difficult to break	
13	[c]6.9×10^{20}	[c]7.4×10^{19}	4.5×10^{19}	1.4×10^{14}	Irregular cracks starting at surface and penetrating into ceramic; ceramic broke when seal was dropped about 2 inches (5.08 cm)	Clean and bright, no indication of corrosion
14	[c]6.1×10^{20}	[c]7.3×10^{19}	4.4×10^{19}	1.3×10^{14}	Irregular cracks similar to seal 13; no dye check	
15	[c]6.0×10^{20}	[c]6.6×10^{20}	4.0×10^{19}	1.2×10^{14}	Irregular cracks similar to seal 13 but not as deep and pronounced; no dye check	
16	[d]4.6×10^{20}	---------	--------	1.1×10^{14}	Irregular cracks similar to seal 13; maximum dye penetration, about 12 percent	
[b]21	[c]5.4×10^{20}	5.6×10^{19}	3.4×10^{19}	1.2×10^{14}	Irregular cracks and some line cracks starting at surface and penetrating into ceramic; ceramic broken before capsule disassembly	
[e]22	[d]3.9×10^{20}	5.1×10^{19}	3.1×10^{19}	1.1×10^{14}	Cracks similar to seal 21; dye test inconclusive because of water inside seal; very difficult to break; see fig. 13	
23 24	[d]3.9×10^{20} [d]3.6	4.8×10^{19} ---------	2.9×10^{19} --------	1.0×10^{14} .9	No indication of cracks; no dye test	

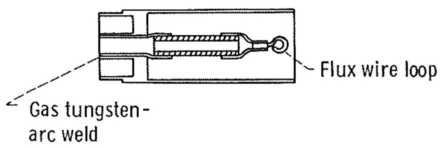
[a]The gamma dose levels are based upon Mockup Reactor measurements converted to Plum Brook Reactor operating levels.
[b]Very small amount of corrosion at center wire penetration into seal end cap (see fig. 12).
[c]These fluence values are based upon nvt measurements from 0.5 percent Co-Al, Ni, Ti, and stainless-steel flux wires. The fast fluences are based upon the normalizations of one-dimensional, 71-group diffusion calculations with the Ni, Ti, and stainless-steel flux wires.
[d]These fluence values are based upon an extrapolation of nvt values of the other seals in a given capsule assembly.
[e]Very small amount of corrosion between center wire and seal end cap (see fig. 14).



(a) Ceramic insulator seal.



(b) Ceramic insulator-seal fixture.



(c) Single seal assembly.

Figure 1. - Single ceramic seal and fixture.

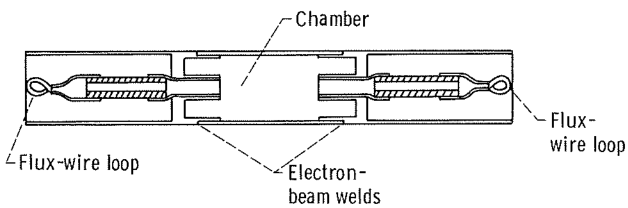


Figure 2. - Double ceramic insulator seal and chamber.

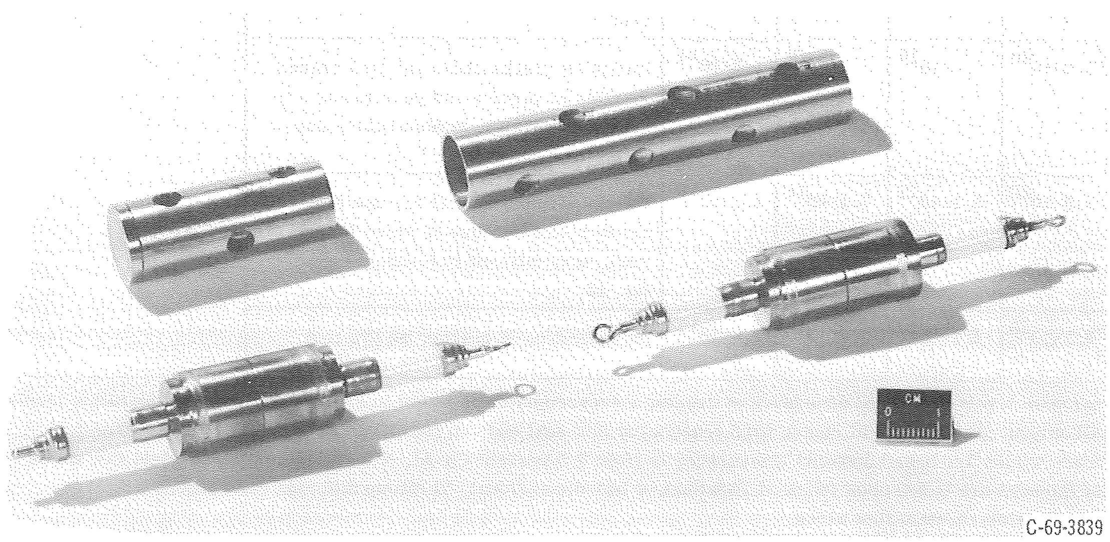


Figure 3. - Ceramic insulator seals and stainless-steel parts which make up one capsule of the multiple-capsule assembly.

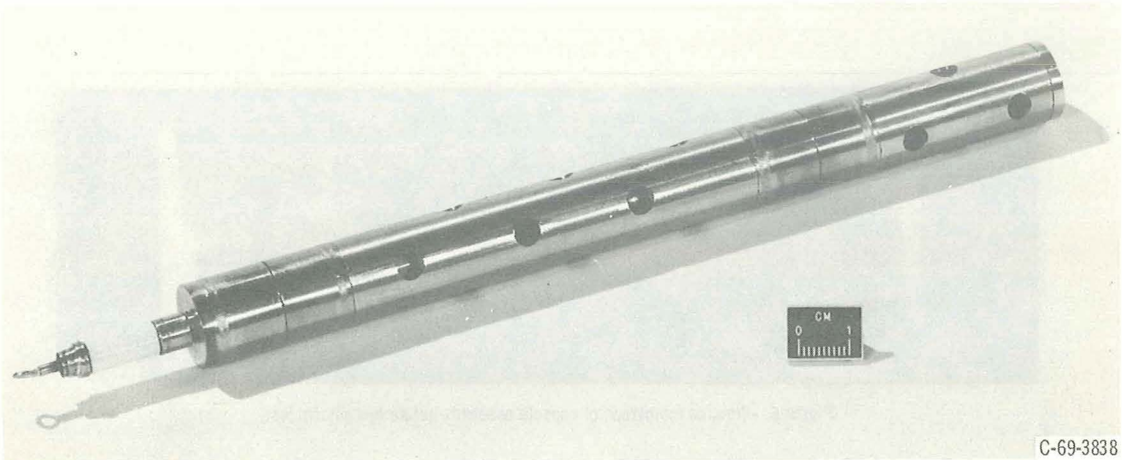


Figure 4. - Ceramic insulator-seal test capsule.

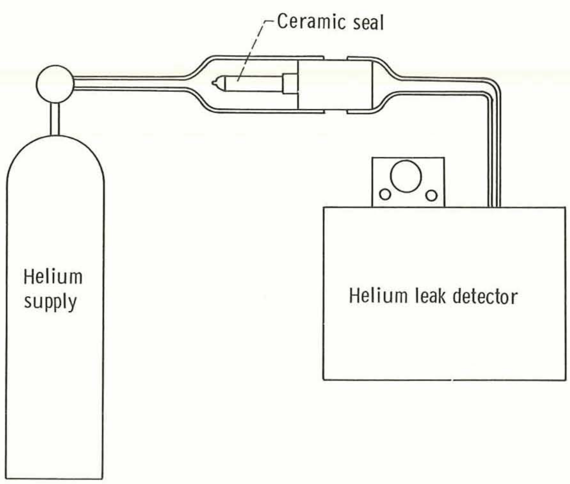


Figure 5. - Apparatus for helium leak-testing of a single ceramic insulator seal during thermal cycling.

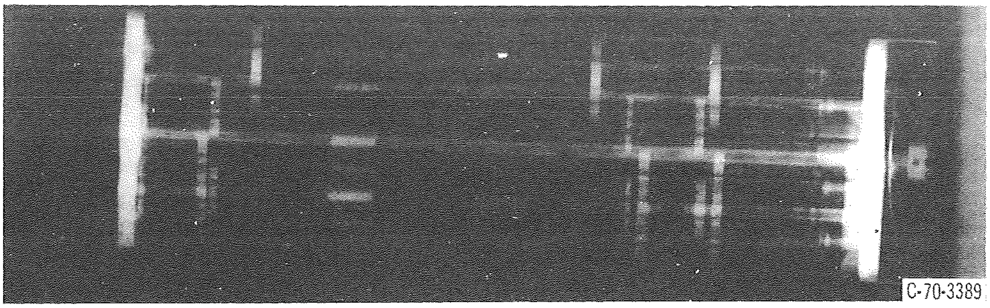


Figure 6. - Neutron radiograph of capsule assembly before hydrostatic test.

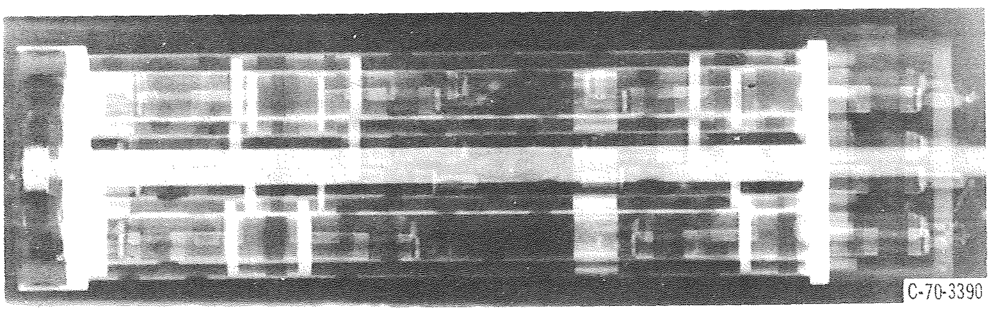


Figure 7. - Neutron radiograph of capsule assembly after hydrostatic test.

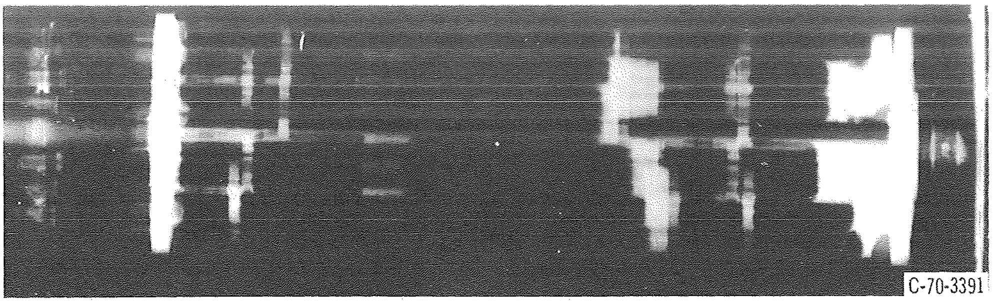


Figure 8. - Neutron radiograph of capsule assembly after 250 hours of irradiation.

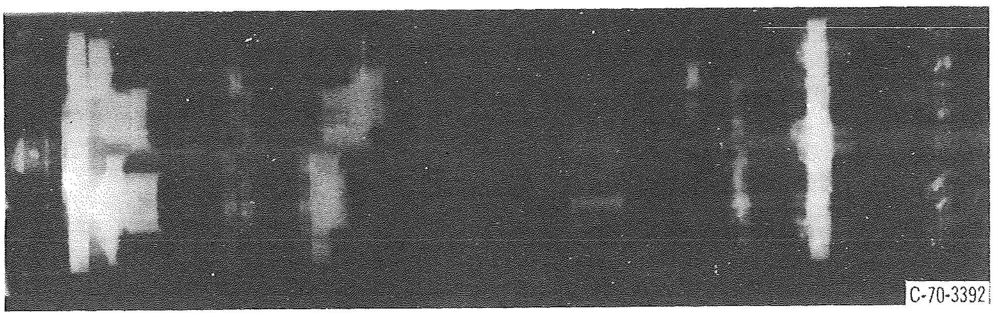


Figure 9. - Neutron radiograph of capsule assembly after 550 hours of irradiation.

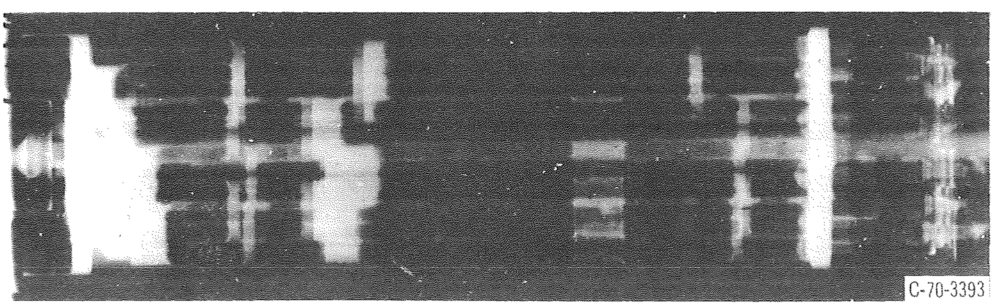


Figure 10. Neutron radiograph of capsule assembly after 960 hours of irradiation.

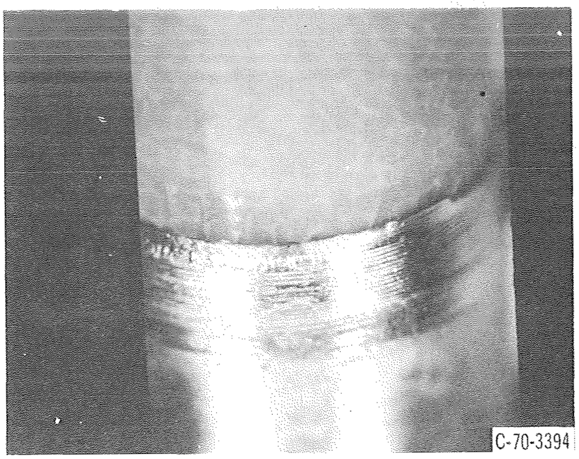


Figure 11. - Seal 5. 2x5.

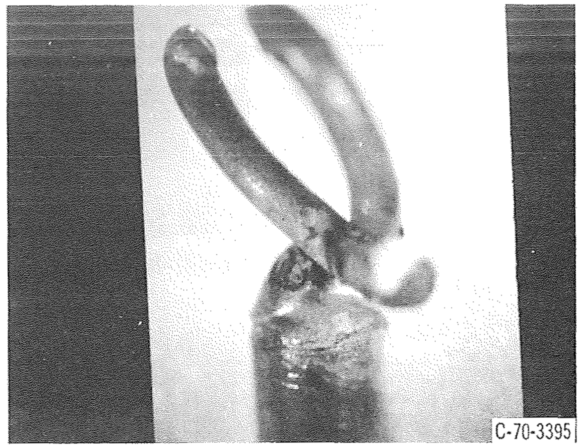


Figure 12. - Seal 5, cap end. 2x5.

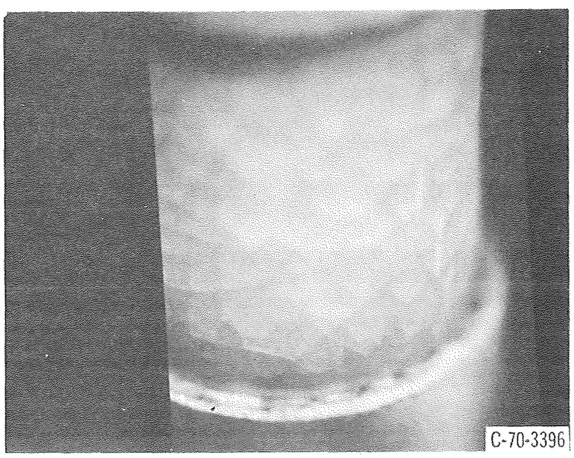


Figure 13. - Seal 22. 2x5.

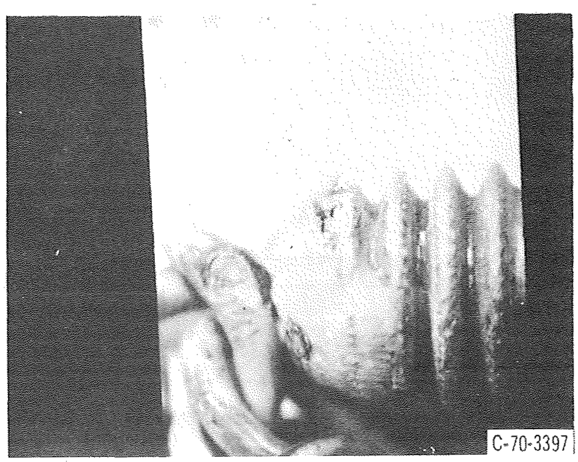


Figure 14. - Seal 21, cap end. 2x5.

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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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